

# Developing a Process for Commercial Silica Production from Salton Sea Brines

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# **Developing a Process for Commercial Silica Production from Salton Sea Brines**

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## Background

The goal of this joint LLNL-CalEnergy project is to develop a method for precipitating marketable silica from spent Salton Sea Geothermal Field (SSGF) brines. Many markets for silica exist. We have initially targeted production of silica as a rubber additive. Silica reinforced rubber gives tires less rolling resistance, greater tear strength, and better adhesion to steel belts.

Previous silica precipitates produced by CalEnergy from Salton Sea brines were not suitable as rubber additives. They did not disperse well in the rubber precursors and produced inferior rubber.

CalEnergy currently minimizes silica scaling in some of their production facilities by acidifying the brine pH. The rate of silica precipitation slows down as the pH is lowered, so that energy extraction and brine reinjection are possible without unacceptable amounts of scaling even with more than 700 ppm  $\text{SiO}_2$  in solution. We are adding a step in which a small amount of base is added to the acidified brine to precipitate silica before reinjection. By carefully controlling the type, rate, and amount of base addition, we can optimize the properties of the precipitate to approach those of an ideal rubber additive.

## Silica as a Rubber Additive

Properties which may be important for rubber additive use include:

1. Surface area
2. Grain size
3. Pore size and pore size distribution
4. Contaminants
5. Surface chemistry (e.g. silanol content and silane coupling)

A commercial silica that is commonly used as a rubber additive is Degussa VN3. We therefore compare the properties of Degussa VN3 with the properties of CalEnergy precipitates and our laboratory precipitates.

## **Bench-top Silica Precipitation Experiments**

We are currently performing bench-top tests of the silica precipitation process using a simplified brine (Na and Ca-chloride) in a 5 liter Teflon-coated glass reaction vessel at 80°C. The optimized process will later be performed on-site in a pilot plant. We start with a 700 ppm silica-containing brine at pH 2, and add a base (NaOH) to raise the pH. To date, we have characterized precipitates from experiments in which the final pH varied from 4 to 8.

## **SEM Photos of Silicas**

SEM photos of silica precipitates show that silica particles are always clusters of smaller silica particles down to the resolution of the SEM. The smallest resolvable particles are about 100-200 nm in diameter. The SEM photos confirm the particle sizes measured using DLS.

(labels for columns of SEM photos)

**Degussa VN3** (SEM#1-3)

**SSGF Region 1** (SEM#4-6)

**SSGF Elmore** (SEM#7-9)

**Laboratory Precipitates** (SEM#10-12)



## Silica-Rubber Bonding

Silicas are treated with a silane coupling agent prior to mixing with rubber precursors. The coupling agent bonds to the silica surface through silanol groups ( $\text{Si-OH}$ ) and changes the silica surface from hydrophilic to hydrophobic. The hydrophobic surface bonds well with the rubber.

The effectiveness of the silica-organic bond is tested by dispersion in oil. Here are micrographs of silane-coupled silicas that have been stirred into polybutadiene, a major rubber component. The particles of our laboratory precipitates initially disaggregated to a greater degree than the Degussa silica. However, further stirring generated Degussa particles that were much smaller than our laboratory precipitates (not shown).

## Particle Size Analysis

Dynamic light scattering (DLS) yields particle size distributions of silica precipitates. With increasing time and pH, our laboratory silica precipitates show growth of larger particles at the expense of smaller particles. Particles appear to grow rapidly and a very short residence time is needed to keep particle sizes small. The size distribution of the pH 5 experiments agrees well with that of the Degussa silica.

## **BET Surface Areas of Silica Precipitates**

CalEnergy silicas have a wide range of surface areas which bracket the surface areas measured for commercial silicas used as rubber additives.

These silicas were sampled from a bench-top experiment in which the pH was increased from 2 to 8 by the addition of NaOH.

## Conclusions

We can produce silicas with many of the same properties as those of commercial silicas.

The oil dispersion tests suggest that our laboratory precipitate does not yet disaggregate to a sufficiently small particle size.

The next step is to send our laboratory precipitate to a tire manufacturer for testing.

Once this work is completed, we will consider designing chemical processes to produce silica for other uses.

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